

DESORPTION OF BURITI OIL (*Mauritia flexuosa*, Mart.) From γ -ALUMINA USING SUPERCRITICAL CO₂

¹Cunha, M.A.E.; ²Machado, N. T*.; ²Araújo, M. E, ³França, L.F.

¹ University of the State of Pará, Center for Natural Science and Technology.

Travessa Enéas Pinheiro 2626, Campus V, Belém-Pará-Brazil.

Phone: 55-091-32769511, Fax: 55-091-237617, CEP: 66095-100

²Laboratory of Separation Processes and Applied Process Thermodynamics, Faculty of Chemical Engineering-UFPA

³Laboratory of Separation Processes, Faculty of Food Engineering-UFPA, Belém-Pará-Brazil

machado@ufpa.br, Fax: 0051-91-32017291

Abstract: In this work, in order to study systematically the enriching of fat-soluble substances by supercritical adsorption/desorption processes, desorption of buriti oil (*Mauritia flexuosa*, Mart.) previously adsorbed in columns packed with γ -alumina has been determined experimentally using supercritical carbon dioxide as solvent. The experiments were carried out in a laboratory scale high pressure extraction unit, adapted to be used as a desorption cell, at 20, and 25 MPa, 333 K, and solvent i rate of $Q_{CO_2} = 10.6$ L/min, using a packed column of 22 cm³ filled with approximately 54-55 grams of γ -alumina + Oil. The results show that buriti oil can be desorbed using supercritical CO₂ and the desorption rate increases with increasing pressure, showing a maximum recovery of 8.53 % [wt.] at 25 MPa.

Keywords: Desorption, supercritical carbon dioxide, vegetable oils, buriti.

1. Introduction

The oil of buriti (*Mauritia flexuosa*, Mart.), a native occurring palm in the Amazon region, obtained by mechanical pressing of pulp, contains between 20-30 % (wt.) of a yellow-orange oil with the highest content of carotenes (~ 3600 ppm) in vegetable oils reported in the literature (França *et al.*, 1999), a natural substance of special interest in the food and pharmaceutical industries.

Despite the development of several processes to recover and enrich carotenes from the palm oil reported in the literature including esterification, and transesterification of palm oil, followed by selective adsorption of the carotenes in packed columns and desorption by using organic solvents (Choo *et al.*, 1991), sterification of palm oil, followed by distillation under high vacuum (Ooi *et al.*, 1991), and adsorption of palm oil within alumina based adsorbents in stirred tanks, followed by supercritical desorption in packed columns using carbon dioxide as solvent (Raiol *et al.*, 2000), no data has been reported concerning the development of methods and or processes to recover and enrich carotenes from buriti oil (*Mauritia flexuosa*, Mart.). Studies reported in the literature concerning the selective separations of substances adsorbed within porous adsorbents by supercritical desorption processes, particularly fat-soluble substances presented in vegetable oils, are scarce. Among those, the desorption of andiroba oil (*Carapa Guianensis*, Aubl.) compounds in packed beds of γ -alumina loaded with andiroba oil at different state conditions (Machado *et al.*, 2005) and fractionation of copaiba oil resin (*Copaifera officinalis*) by supercritical desorption in packed columns with γ -Alumina as adsorbent (Machado *et al.*, 2009).

In this work, in order to develop a data basis to study systematically the performance of γ -alumina to selectively fractionate and enrich fat-soluble substances present in buriti oil,

particularly carotenes and tocopherols, desorption of buriti oil from γ -alumina using SC-CO₂ has been investigated in a laboratory scale high pressure extraction unit, adapted to be used as a desorption cell, at 20, and 25 MPa, 333 K, and solvent flow rate of $Q_{CO_2} = 10.6$ L/min.

2. Materials and Methods

2.1 Materials - Aluminum hydroxide used as raw material for the adsorbent preparation was delivered by Alunorte S/A (Barcarena-Pará-Brazil). Carbon dioxide 99.90 % [vol/vol] pure was supplied by Linde Gases Ltda (Belém-Pará-Brasil). Buriti oil (*Mauritia flexuosa*, Mart.) was obtained by exhaustive supercritical carbon dioxide extraction of buriti pulp at 25 MPa and 333 K using a SFE unit described elsewhere (França *et al.*, 1999).

2.2 Absorbent Preparation and Characterization - Aluminum hydroxide Al₂O₃ (*Gibbsite*), the precursor of γ -alumina has been obtained by the methodology described elsewhere (Machado *et. all*, 2009). The characterization of γ -alumina is depicted in Table 1.

Table 1: Particle average pore diameter (ϕ), particle porosity (ϵ_p), particle mean sauter diameter (d_p), and particle specific area (a_p) of Boehmite and γ -alumina.

Alumina	ϕ [nm]	ϵ_p [-]	d_p [μ m]	a_p [m ² /g]
Boehmite	5.535	0.210	118.77	151.92
γ -Alumina	7.883	0.305	141.52	153.79

2.3 Experimental Apparatus – A schematic diagram of the high pressure apparatus used in this work is depicted in Figure 1. The unit consists of high pressure vessel of 1000 cm³, adapted to be used as a desorption cell, one cylinder of 22 cm³, a membrane compressor, a CO₂ reservoir, a sampling system, a gas meter, for more details see (França *et al.*, 1999).

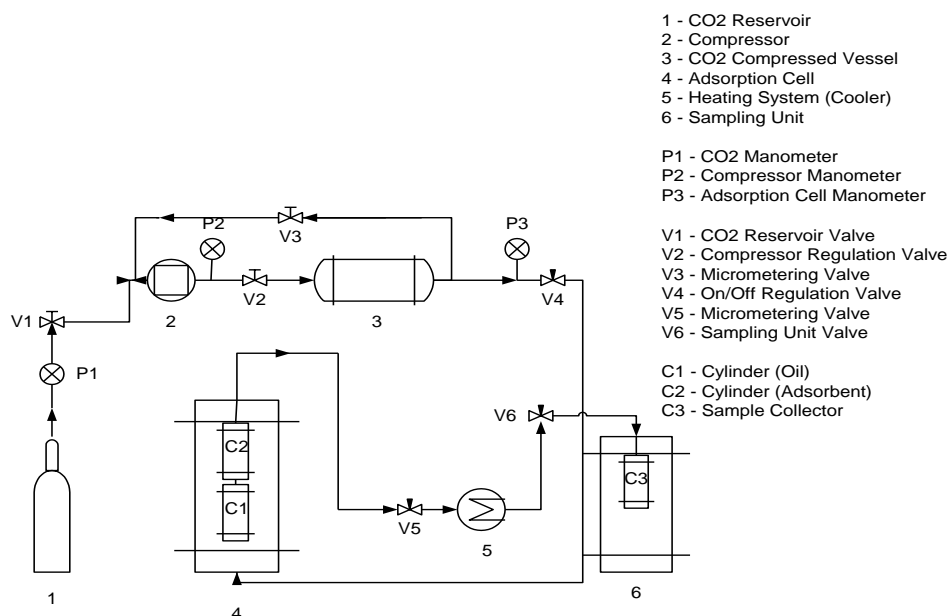


Figure 1: Experimental set-up of the high pressure desorption unit.

2.4 Experimental Procedure (Supercritical Desorption) – The desorption experiments were carried out using approximately 54-55 g of γ -alumina loaded with buriti oil. γ -alumina loaded with buriti oil was placed inside the upper cylinder of 22 cm³ (C2) depicted in Photography 1.



Photography 1: Stainless steel cylinder of 22 cm³.

The cylinder has 02 (two) flanges, whereas 03 (three) slices of filter paper between 02 (two) external stainless steel screens of mesh 200# were placed in order to avoid the adsorbent particles loaded with buriti oil to spread out the upper cylinder.

The carbon dioxide was recycled in a closed loop for at least for 20 (twenty) minutes, by closing valves V₅ and V₆, and opening valves V₁, V₂, V₃ (micrometer/recycle valve), and V₄, in order to maintain the system pressure constant. Afterwards, valves V₅ and V₆ were open and the flow rate was set low (9.6-10 g_{CO2}/min). The low flow rate makes it possible the fresh carbon dioxide that enters the high pressure vessel to recover the oil adsorbed in γ -alumina. Samples from the gaseous phase were taken every 05 (five) minutes by opening valves V₅ and V₆. The condensed phase was weighted by gravimetric method. The CO₂ released into the atmosphere was measured using a gas flow meter. Since, ambient pressure and temperature are measured at gas meter inlet, the density of carbon dioxide can be computed using the bender equation of state. The desorption kinetics is computed by the cumulative mass of oil versus time. The total time interval for the desorption experiments carried out at 333 K, and pressures of 20, and 25 MPa were 300 and 195 minutes respectively.

2.5 Desorption Kinetics – The desorption kinetics is computed by the cumulative mass of buriti oil versus time for the desorption experiments using equation (1) as follows.

$$M_{\text{Oil Desorbed}} = \sum m_{\text{Condensates}}(t) * \Delta t \Big|_{\text{Desorption}} \quad (1)$$

Where $\sum m_{\text{Condensates}}(t) \cdot \Delta t \Big|_{\text{Desorption}}$ is the cumulative mass of condensates weighted within the sampling intervals of time during the desorption experiments, Δt is the total time interval. Equation (2) shows a mass balance of buriti oil adsorbed in γ -alumina for the desorption process in supercritical media.

$$M_{\text{Buriti Oil in } \gamma\text{-alumina}} \Big|_{(0)} = M_{\text{Buriti Oil in } \gamma\text{-alumina}} \Big|_{(t)} + \sum m_{\text{Condensates}}(t) \cdot \Delta t \Big|_{\text{Adsorption}} \quad (2)$$

Equation (2) shows that initial mass of buriti oil adsorbed in γ -alumina is the sum of remaining mass of buriti oil in γ -alumina and the mass of buriti oil desorbed for $t > 0$. In all cases, the mass balance closure delivered quite exact results.

3. Results and Discussion

The desorption experiments of γ -alumina loaded with buriti oil using supercritical CO_2 as solvent were carried out at 20, and 25 MPa, 323 K, and solvent flow rate of $Q_{\text{CO}_2} = 10.6$ L/min. The desorption kinetics is shown in Figure 2. The experimental results show that the desorption rate increases as pressure increases, showing a maximum recovery of 8.53 % [wt.] at 25 MPa, and that supercritical carbon dioxide is able to recover buriti oil adsorbed in γ -alumina.

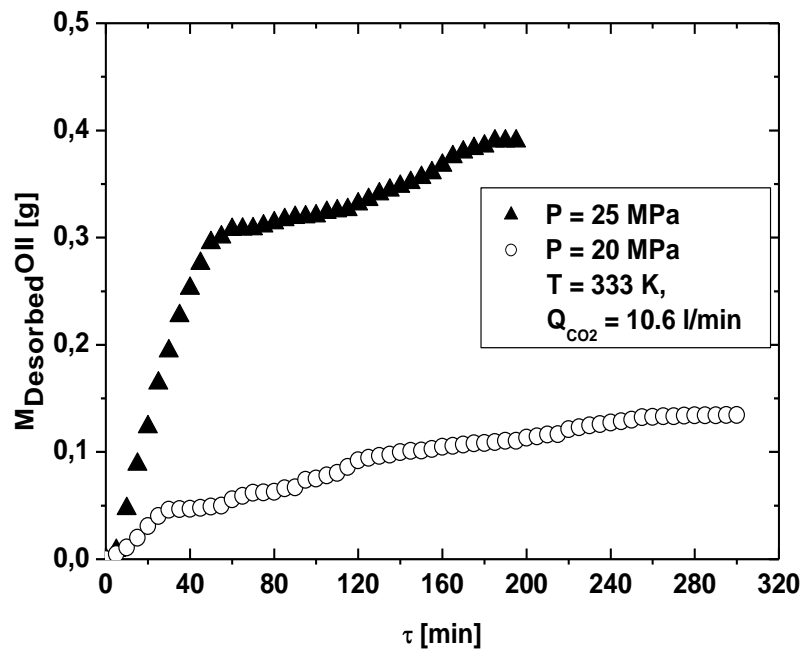


Figure 2: Desorption kinetic of oil buriti in using SC- CO_2 at 20, and 25 MPa and 333 K.

Table 2 shows the capacity of γ -alumina used on the desorption experiments using supercritical CO₂ as solvent and the total mass of buriti oil desorbed.

Table 2: Capacity of γ -alumina used on the desorption experiments using supercritical CO₂ as solvent and the total mass of buriti oil desorbed at 20, and 25 MPa and 333 K.

P [MPa]	T [K]	Mass γ -alumina + Oil [g]	Mass Oil [g]	Capacity [mg _{Oil} /g _{γ-alumina}]	M _{Oil Desorbed} [g]
20	333	54.9743	4.9681	99.35	0.1343
25	333	54.9743	4.9681	99.35	0.4171

4. Conclusions

The experimental results show that the desorption rate increases as pressure increases, showing a maximum recovery of 8.53 % [wt.] at 25 MPa, and that supercritical carbon dioxide is able to recover buriti oil adsorbed in γ -alumina, in order to enrich fat-soluble substances.

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